



**AFRL-RQ-WP-TR-2020-0081**

**REPLACEMENT OF OBSOLETE TUBE DEPOSIT RATER  
(TDR) WITH ELLIPSOMETER FOR JET PROPELLANT  
THERMALLY STABLE (JPTS) (MIL-DTL-25524)  
CONFORMANCE**

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**OCTOBER 2020  
Interim Report**

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## **1.0 EXECUTIVE SUMMARY**

This report contains a summary of the project to validate the use of the ellipsometric tube (deposit) rater (ETR) in place of the obsolete/unsupported tube deposit rater (TDR) in the JPTS specification MIL-DTL-25524. A corollary effort was conducted to validate the use of the interferometric tube rater (ITR) as well. Both the ETR and ITR were found to be a suitable replacement for the TDR, and the transition partner (Air Force Petroleum Agency) proceeded with a plan to replace all of the TDRs with ETRs. The project was funded by the AFRL Sustainment Office (PE 63199F/61102F).

## 2.0 BACKGROUND

The specialty kerosene fuel JPTS (Jet Propellant Thermally Stable, MIL-DTL-25524) was developed for the U-2 beginning in 1956 [Martel, 1987]. Because of the U-2's flight altitude and other factors, the thermal stability of JPTS was increased over conventional jet fuels, while the freeze point was decreased. JPTS is still in use today, with the current specification version being MIL-DTL-25524G. The thermal stability requirement is defined by ASTM D3241: Standard Test Method for Thermal Oxidation Stability of Aviation Turbine Fuels. [Note: ASTM D3241 is often referred to as the JFTOT; however, JFTOT™ is the registered trademark name for the D3241 apparatus sold by Alcor (now owned by PAC). This report uses the term JFTOT as a generic reference to the instrument/apparatus prescribed by method D3241 and not exclusively the instrument brand.] In the ASTM D3241 method, jet fuel flows through an annulus for 2.5 hours, with the inner portion of the annulus being a heated aluminum tube. The fuel exits the heated portion and passes through a 17 micron (nominal) stainless steel filter; the pressure drop across the filter is monitored as a test metric. For JPTS, the tube temperature setting is 335 °C; for conventional jet fuels, the temperature is 260 °C, illustrating the higher thermal stability requirement. The result of the test is a thermal-oxidative deposit on the tube. The deposit is rated (either by color code or by thickness) and these values are used as metrics. The limits of both deposit rating and pressure drop are defined by the jet fuel specification.

One of the primary difficulties with the JFTOT is measuring the amount/thickness of deposits on the polished aluminum tubes. Throughout most of the last 30 years, the deposits on the JFTOT tubes were rated visually and compared to a set of color standards (ASTM D3241, Annex 1). This technique was carried over from the predecessor of D3241, i.e., ASTM D1660 (aka the ASTM-CRC Fuel Coker). Over the years, this visual rating method has been criticized for various reasons, e.g., being sensitive to human subjectivity; thus, there have been efforts over the years to develop improved rating methods. As early as the mid-1970s, there were efforts to develop quantitative rating techniques for JFTOT heater tubes, which was about the same time D3241 started to replace D1660 (CRC Project CA-43-67, 1974; Martel & Bradley, 1975). Commercial aviation seemed content to use the visual rating method; however, the military adopted the more advanced tube deposit rater (TDR) technique for JPTS and JP-7 fuel as described by Martel (1987):

“The Alcor Corporation, developer of the JFTOT, developed a Tube Deposit Rater (TDR) that measures the change in heater tube reflectivity caused by the deposits. An unresolved controversy exists as to the use of the TDR, as the visual rating and the TDR often give conflicting results. However, there has always been a problem with the visual rating method, as some deposits of different colors and that color standards, and deposits may be dull or glossy. Other tube deposit rating techniques under consideration include beta-ray backscatter, light interference, voltage breakdown through the deposit, and deposit burnoff.

For JPTS and JP-7 fuels, the JFTOT test method and the Alcor TDR deposit rating method are specified. To further improve the tube deposit rating procedure, the JFTOT heater tube is rated both before and after the test, and the maximum increase caused by the deposits is the criterion used. This method has shown good precision and removes the subjectivity of the visual rating method.”

That was the situation in 1987. Subsequently, the majority of D3241 heater tube evaluations for conventional fuels used the visual rating method, and the use of the TDR was confined mainly to JPTS and JP-7. This small market caused Alcor to stop producing and supporting the TDR, to the

point in 2014 at which the cost of individual TDR maintenance and calibration was \$1,525 per unit per year. Calibration of TDRs became an annual requirement for the Air Force Petroleum Agency.

Commercial instrument manufacturers had made progress in developing alternatives to both the visual rating and TDR methods. In 2014, two new annexes to D3241 were approved that allowed for interferometric and ellipsometric means of rating heater tubes. Both techniques use a light source with a small spot size (approximately 200 micron) to determine the absolute thickness of deposit at 1,200 points on the heater tube. These data allow for a three-dimensional map of deposit thickness on a heater tube. For specification purposes, this extensive amount of data was reduced to a single deposit thickness metric: the standard spot. The technical challenge for the fuels community was now how to transition the outdated rating techniques, contained within jet fuel product specifications, to the new metrics with equivalent performance. ASTM conducted studies to provide guidance regarding the transition from the visual tube rating method (VTR) to the two metrological methods: ellipsometric tube rating (ETR) in Research Report RR: D02-1774 and interferometric tube rating (ITR) in Research Report RR: D02-1786. The consensus was that a maximum deposit thickness limit of 85 nm (by either ETR or ITR) was equivalent to the visual limit of less than color code 3 (by VTR). *However, no guidance was given regarding the transition of TDR limits to deposit thicknesses by ETR or ITR.*

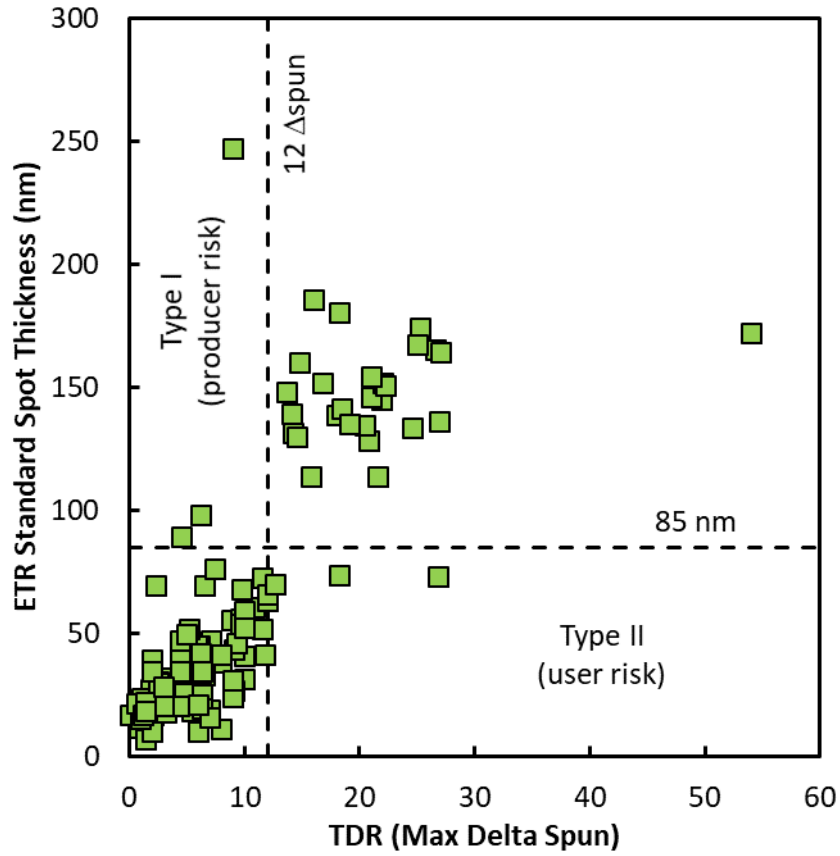
It was at this point that the Air Force Research Laboratory's Fuels Branch, the University of Dayton Research Institute, and the Air Force Petroleum Agency collaborated on a proposal to the AFRL Sustainment Office to fund the necessary program to demonstrate that the modern ellipsometer and/or interferometer could reliably replace the outdated and unsupported TDR in assessing the thermal stability of JPTS. This report describes the results of that program.

### **3.0 EXPERIMENTAL**

Heater tube specimens were generated via ASTM D3241 protocol with various batches of JPTS grade fuel. Most of the specimens were generated under MIL-DTL-25524 prescribed operating conditions, i.e., 335 °C set point temperature and 2.5 hour duration; however, some specimens were generated at alternative temperatures less than 335 °C. Regardless, surfaces of the heater tube specimens were evaluated using the following three instruments: an Alcor Mark 8A TDR, a Falex model 430 ETR, and an AD Systems model DR-10 ITR.

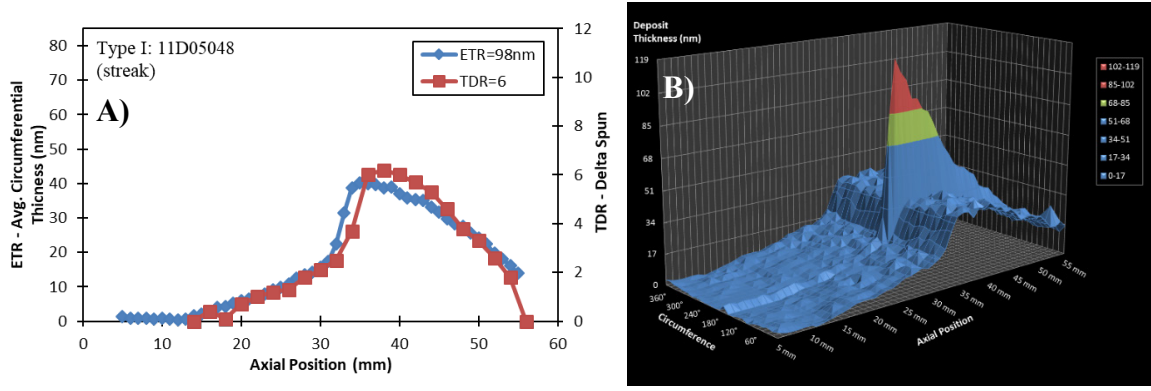
#### 4.0 RESULTS AND DISCUSSION

A total of 178 heater tube specimens were generated over a period of about 18 months using various batches of JPTS fuel. The heater tubes were generated at different laboratories and while a best effort was taken to evaluate each tube using as many rating methods as possible, i.e., TDR, ETR, and ITR, that was not always the case (see the Appendix for all data). Nevertheless, method-to-method comparisons were made using not less than 93 samples. Figure 1 shows a direct comparison of tube ratings using the ETR versus the TDR. The ETR reports the maximum spot thickness—that is the greatest average deposit thickness over a 2.5 mm<sup>2</sup> area (per ASTM D3241)—while the TDR provides the maximum  $\Delta$ spun. In theory, the  $\Delta$ spun rating correlates to a deposit thickness since it measures the difference in light reflected from the circumference of the tube before and after being exposed to fuel stressing; that is to say large  $\Delta$ spun values correlate to less light reflected (i.e., more light absorbed by the deposit) and therefore thicker deposits. Dashed lines at 12  $\Delta$ spun and 85 nm are shown for reference, i.e., the TDR specification limit for JPTS and the ETR specification limit for Jet A, respectively. Figure 1 shows that both heater tube evaluation techniques trend similarly. The Pearson correlation coefficient for these data is computed to be 0.796, which means a positive correlation is observed between ETR standard spot thickness and TDR max  $\Delta$ spun. In fact, out of the 123 heater tubes assessed only 6 heater tubes (less than 5%) showed either type I (3 heater tubes) or type II errors (3 heater tubes). A type I error is defined as an assessment that would have been accepted using the incumbent TDR method, however, is rejected using the candidate ETR method; these errors are considered a risk to the producer as the fuel would be rejected using the new evaluation technique. Type II errors are defined as an assessment that would have been rejected using the incumbent TDR method, however, are accepted using the candidate ETR method; these errors are considered a risk to the user as the fuel might be of lesser quality, however, would be accepted using the new evaluation technique.



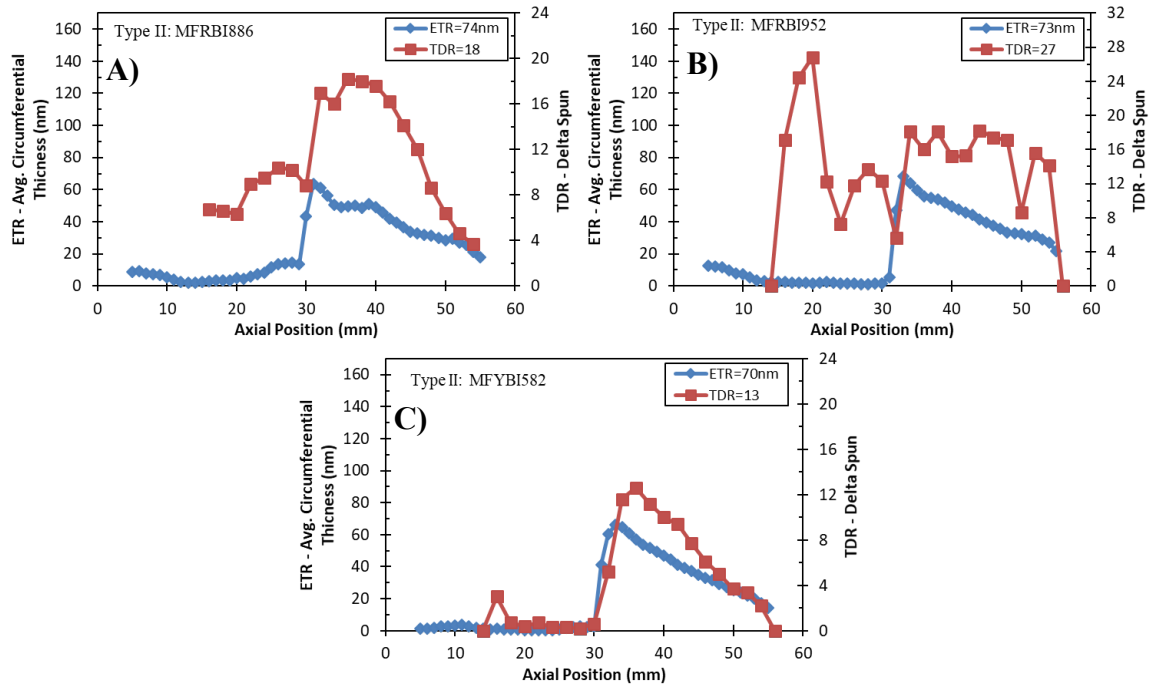
**Figure 1.** ETR standard spot thickness versus TDR  $\Delta$ spun for 123 heater tube specimens.

Further investigation into some of the heater tubes exhibiting type I and type II errors, reported in Figure 1, provides insight about both evaluation techniques. Figure 2 shows deposition profiles of a tube that exhibited a type I error. As the figure shows, the average circumferential profiles for both the ETR and TDR are well matched. However, upon inspection of the full deposit surface topography—using the ETR—a long, narrow deposit (aka a streak) is predominant. This deposit streak was significant enough to be seen by the naked eye. While the error is considered a producer risk, the presence of such a large deposit could have caused a risk to the user. Therefore, the ETR provided a more discerning and conservative result.



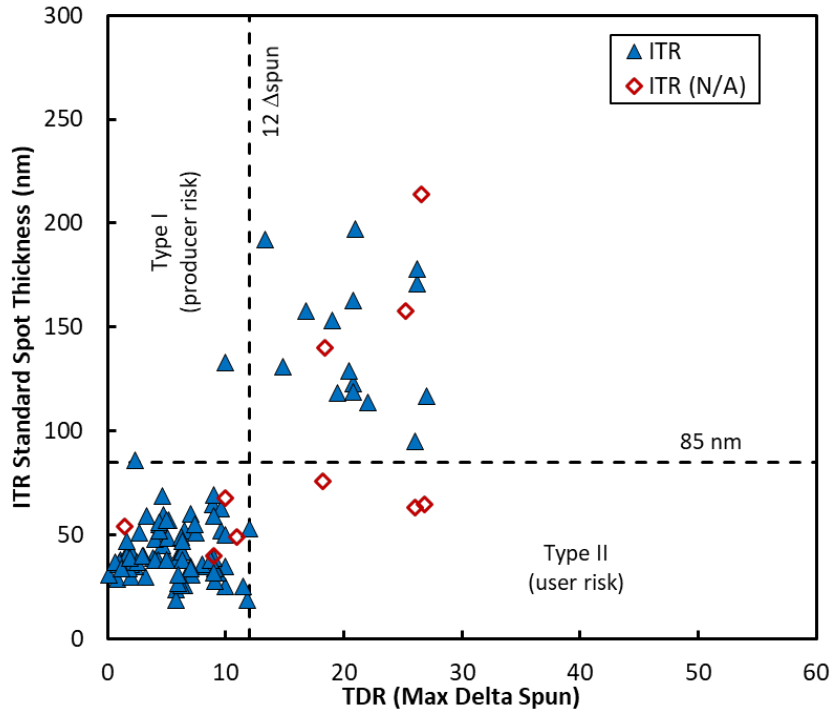
**Figure 2.** Tube exhibiting type I error due to deposit streak, max ETR and max  $\Delta$ spun TDR values shown in legend; a) TDR and ETR circumferential deposit profiles, fuel flow from left to right, b) full heat tube deposit topography using ETR.

Figure 3 shows deposit profiles, measured using both the TDR and ETR, of heater tube specimens exhibiting type II errors. It can be seen in Figure 3A that the TDR gives elevated deposit values early in the tube, from about 15 to 30 mm in axial position. From prior experience, it is unlikely that deposits are forming this early in the tube, therefore, it is believed that the TDR is providing an erroneously high baseline for this case, which skews values above the pass/fail line of 12. The TDR response in Figure 3b exhibits a large variance; therefore, there is low confidence in the absolute values reported for this specimen and the ETR data is preferred. Results shown in Figure 3c demonstrate good correlation between measurement techniques, the only difference between TDR and ETR data appears to be a slight difference in response factor (that is the TDR reports slightly more deposit than the ETR). With the data well matched in this last case, the TDR provides a realistic, more conservative assessment. Even though type II errors are considered user risk, two out of these three cases (Figures 3a and 3b) tend to favor the results from the ETR over the TDR with respect to reliability and confidence in the measurement.



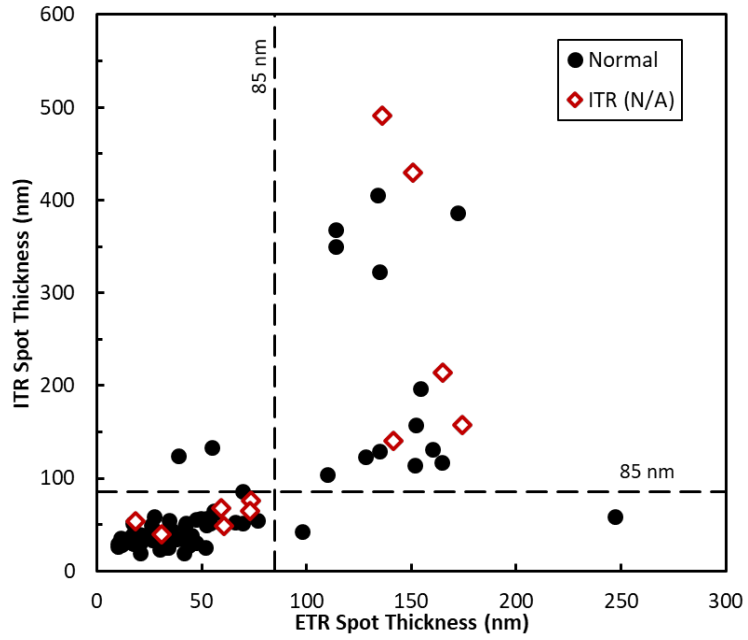
**Figure 3.** Heater tube specimens exhibiting type II errors.

In addition to the ETR, the ITR was used to evaluate heater tube specimens and results were compared directly to TDR data. Figure 4 shows the direct comparison of ITR results to TDR results for 118 heater tubes. As the figure shows, a positive correlation is observed between ITR and TDR results. The Pearson correlation coefficient for these data is 0.718. Only five of the heater tubes (or about 4%) exhibit either a type I (two heater tubes) or type II (three heater tubes) error. The ITR instrument reports N/A when it encounters a measurement datum that appears to be an outlier according to the algorithms used to determine thickness from optical properties. The ITR instrument will continue to report thickness values for the remaining portions of the tube, however, once an N/A datum is reported the resulting heater tube evaluation is considered a fail according to the manufacture. These N/A data are shown as red diamonds in the figure. When considering the N/A points as failures, this increases the number of type I errors to six heater tubes (or about 5% of the tested samples) and decreases the number of type II errors to zero. This results in a more conservative evaluation for the user.



**Figure 4.** ITR standard spot thickness versus TDR  $\Delta_{spun}$  for 118 heater tube specimens.

As an additional check on the methods used, 93 heater tubes were analyzed using both the ETR and ITR methods; these results are shown in Figure 5. As the figure shows, a positive correlation is exhibited between the results of the two rating methods with a Pearson correlation coefficient of 0.672. While there is significant variance of the absolute thickness for deposits greater than about 100 nm, for the sake of evaluating specimen for jet fuel specifications both instruments agree with respect to the pass/fail criterion of 85 nm at these elevated deposit thicknesses. Out of the 93 heater tubes evaluated there is pass/fail disagreement for 11 tubes, although 6 of the conflicting tubes are due to N/A evaluations from the ITR. However, the primary purpose of this work was not to compare ETR to ITR results since these two methods are already approved evaluation techniques. Rather, the intention of Figure 5 is to demonstrate that agreement between the two approved metrology techniques is in line with the agreement seen between the TDR-ETR and TDR-ITR results.



**Figure 5.** Parity plot of standard spot thickness between ITR and ETR for 93 heater tubes.

## 5.0 CONCLUSIONS

Heater tube specimens were evaluated using the TDR, ETR, and ITR techniques. Results show that positive correlations exist for all three parity combinations, i.e., ETR-TDR, ITR-TDR, and ITR-ETR. Correlation between the TDR and both the ETR and ITR appear to be within acceptable tolerances. The existing ASTM D1655 specification limit of ETR or ITR  $\leq 85$  nm is recommended to replace the MIL-DTL-25524 limit of TDR  $\leq 12$   $\Delta$ spun. This proposed modification to the MIL-DTL-25524 specification simplifies implementation while maintaining a larger degree of severity for JPTS conformance due to the higher D3241 operating temperature, i.e., JPTS operating temperature requirement of 335 °C and Jet A operating temperature requirement of 260 °C.

## **6.0 REFERENCES**

Martel, C. R., "Military Jet Fuels, 1944-1987," AFWAL-TR-87-2062, 1987.

Martel, C. R., Bradley, R. P., "Comparison of Rating Techniques for JFTOT Heater Tube Deposits," AFAPL-TR-75-49, 1975.

## Appendix

Table A1. Combined Results of Heater Tube Evaluations

Tube S/N	VTR Code	$\Delta P$ (mmHg)	TDR (max $\Delta$ spun)	Standard Spot Thickness (nm)		ITR 'N/A' Code
				ETR	ITR	
LIGAE225			0	17	31	
MFOBI469			1	21	37	
LHdAE250			1	12	29	
ME4BI757			1	15	36	
MFOBI307			1	24	37	
MEXBI413			1	18	38	
MFRBI523			1	17	34	
LHZAE247			1	7		
MFRBI764			1	22		
MFYBI627			1	18	54	
MEXBI466	1A	0	2		40	
ME4BI032	1		2		37	
LHaAE080			2	16		
LHKAE100			2	11		
MFRBI725	2		2		47	X
MEXBI689	1A	0	2		40	
LHaAE102			2	19	36	
ME4BI738	1		2	27		
MFOBI374	2A	0	2		34	
ME4BI524	1		2	25		
MFRBI878			2	34	39	
LEBAB495		0	2	23	36	
LFVAC073			2	26	40	
MBVAG340			2	10	30	
MBVAG572			2	22	39	
ME4BI198	1		2	28		
ME4BI285		0	2		42	
ME5BI118			2	39	36	
100512A-00051			2	17	40	
LEBAB209			2	23		
LHKAE266			2	24	38	
LEBAB372			2	70	86	
LHaAE107			2	18		
ME4BI192	1		2		35	
101020A-00076			3	24		
LHdAE183			3	24		
ME4BI688	1		3	30		
MFRBI333			3	30	37	
MFRBI415	1A	0	3		51	
MFRBI909			3	29	40	

Table A1 (continued). Combined Results of Heater Tube Evaluations

Tube S/N	VTR Code	$\Delta P$ (mmHg)	TDR (max $\Delta$ spun)	Standard Spot Thickness (nm)		ITR 'N/A' Code
				ETR	ITR	
LhaAE173			3	32		
MEXBI321			3	20	40	
MBVAG456			3	18	30	
100408A-00013			3	27	59	
ME5BI354	2A	0	4		39	
ME4BI926		0	4		48	
MJBB1296	<1	0	4		38	
ME4BI180	<1	0	4	21	38	
MFRBI508			4	42	52	
ME5BI845	1A		4	34	55	
MFRBI628			4	47	56	
LIdAE519	<1	0	5	89		
LEBAB552			5	35	45	
MFOBI355	1A	0	5		69	
LHZAE357			5	36		
LEBAB063			5	26		
MFOBI457	1A	0	5		60	
MFRBI874			5	50	57	
MJIB1106	<1	0	5		38	
MEXBI874	2A	0	5		49	
13A03312		0	5	42	39	X
MBVAG546	2		5	52	57	
LHXAE553			5	18		X
11D05024		0	6	41		
13A03311		0	6	21	19	
11D05039		0	6	30	24	X
LIZAE288			6	45		
LHOAE070	1		6	10	27	
LHVAE348	<1		6	20	39	
MJBB1065	1		6	21	31	
11D05045		0	6	45	39	
13A03320		0	6	34	26	
11D05048		0	6	98	43	
MFRBI977			6	42	49	
MFRBI097	<2		6	25	42	
MFRBI712			6	19	38	
MHMBI497			6	35	47	X
11D05040		0	7	69	52	
11D05052		0	7	33	26	
100518A-00299			7	45		

Table A1 (continued). Combined Results of Heater Tube Evaluations

Tube S/N	VTR Code	$\Delta P$ (mmHg)	TDR (max $\Delta$ spun)	Standard Spot Thickness (nm)		ITR 'N/A' Code
				ETR	ITR	
LHXAE526	1	0	7	19	35	
ME4BI464		0	7		60	
MITBI665	1	0	7	16	34	
11D05031		0	7	47	31	
MFRBI336	2A		7	76	55	
MFRBI320	<3A	0	8		51	
11C09800	1	0	8	38	35	
LHOAE205	1		8	41	35	
LHXAE218	1	0	8	11	36	
MFRBI923	1A		9		38	
11D05022		0	9	56	65	
LHOAE427	1	0	9	24	39	
MBVAG442	3A	0	9		69	
ME4BI514			9	31	40	
ME5BI867			9	247	59	
MJIBI238	<1		9		32	
LEBAB509			9	27	34	
11D05032		0	9	44	28	
13A03319		0	9	46	32	
11D05027		0	10	56	63	
ME5BI314	2		10	54	52	
LIGAE162			10	68		X
11C09781	1	0	10	32	26	
11C09798	1	0	10	41	35	
MBVAG595			10	55	133	
MFRBI888	2A		10	59	68	
MHMBI206		0	10	52	50	
11D05037		0	11	61	49	
11D05025		0	12	52	25	
LIGAE244			12	73		
11D05021		0	12	41	19	X
LIGAE234			12	63		
MFRBI303	<3A		12	66	53	
MFYBI582			13	70		
MEXBI745	>4A	0	13		192	
LIGAE005			14	148		
LHKAEE263			14	139		
LHaAE106			14	132		
LhaAE146			15	130		
MFOBI157	4	0	15	160	131	

Table A1 (continued). Combined Results of Heater Tube Evaluations

Tube S/N	VTR Code	$\Delta P$ (mmHg)	TDR (max $\Delta$ spun)	Standard Spot Thickness (nm)		ITR 'N/A' Code
				ETR	ITR	
MFRBI565			16	114	368	
MFYBI155			16	186		
MFRBI440			17	152	158	
MFRBI730			18	139		
MFRBI786			18	181		
MFRBI886			18	74	76	
MFRBI752			18	141	140	
MFOBI406			19		153	
MFRBI758			19	135	323	
MEXBI969	>4A	0	20		118	
MFRBI143	>4A		20	135	129	
MFRBI034	4A		21	128	123	
MFRBI441			21		119	
MFRBI441			21		163	X
LIGAE018			21	146		
MFRBI823			21	154	197	
MFRBI416	>4A		22	114	350	
LHaAE285			22	145		
ME5BI728	>4A		22	152	114	
MFRBI057	>4A/P		22	151	430	
MFRBI351	>4A		25	134	405	X
MEXBI102			25	168		X
MBVAG498	>4A		25	174	158	
MFOBI285			26		63	
MFOBI285			26		95	X
MFRBI117			26		171	
MFRBI117			26		178	
MBVAG398	>4A		27	165	214	
MFRBI952			27	73	65	X
MBVAG217	>4A/P		27	136	491	
MBVAG658			27	165	117	
MFRBI122	>4A/P		54	172	386	
ME5BI136	1	4		17	52	
ME5BI170	2	38		39	124	
ME5BI345	2	21		26	50	
ME5BI372	<4A	33		110	104	